A Study of the Self-Passivation of Space-Survivable POSS Kapton Polyimides

Dr. Sandra J. Tomczak
AFRL/PRSM Materials Application Branch
Space and Missile Propulsion Division
10 East Saturn Blvd., Bldg. 8451,
Edwards AFB, CA 93524-7680
Phone: (661) 275-5171
sandra.tomczak@edwards.af.mil

Mrs. Vandana Vij, Dr. Tim Haddad, ERC Inc.
Dr. Darrell Marchant, Dr. Joe Mabry (Group Lead)
AFRL/PRSM, Edwards AFB, CA.

Dr. Timothy K. Minton, Amy Brunsvold, Montana State University

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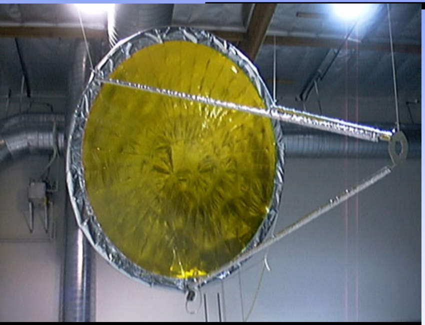
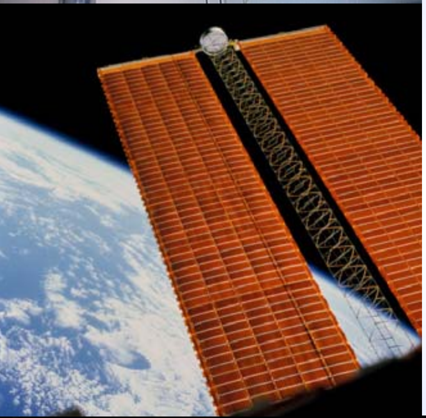
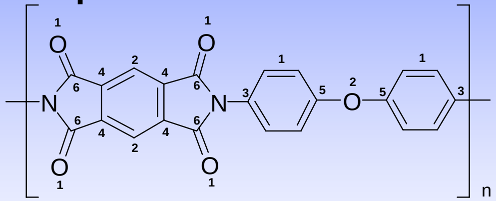
Goal



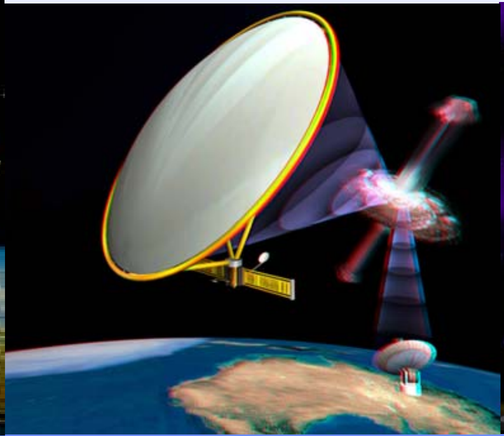


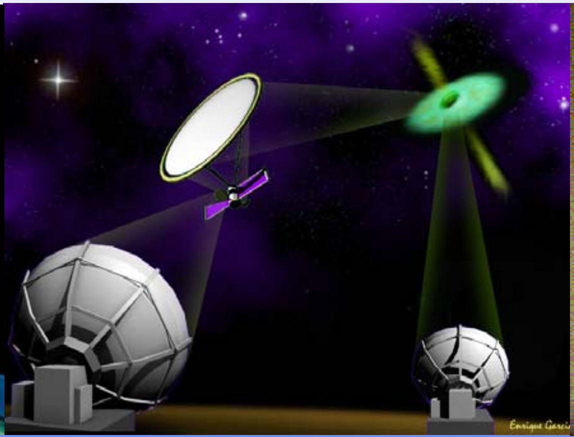
Kapton Structure

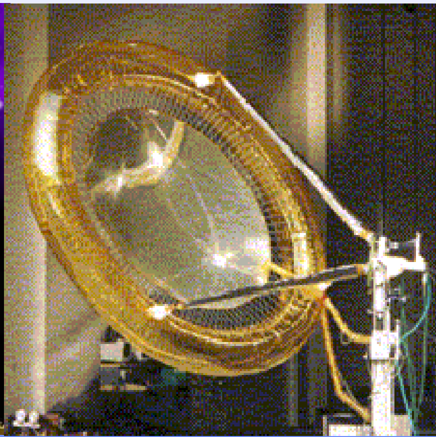


Our goal is to create an efficient drop-in replacement for Kapton that:

1. Has increased space survivability due to **resistance to atomic oxygen**, thermal cycling, solar UV and VUV radiation, protons and electrons.
2. Is **Self-Passivating** based on hybrid organic/ inorganic nanocomposite incorporation
3. Has superior optical properties, low solar absorptance, high thermal reflectance
4. Has excellent mechanical thermal properties.







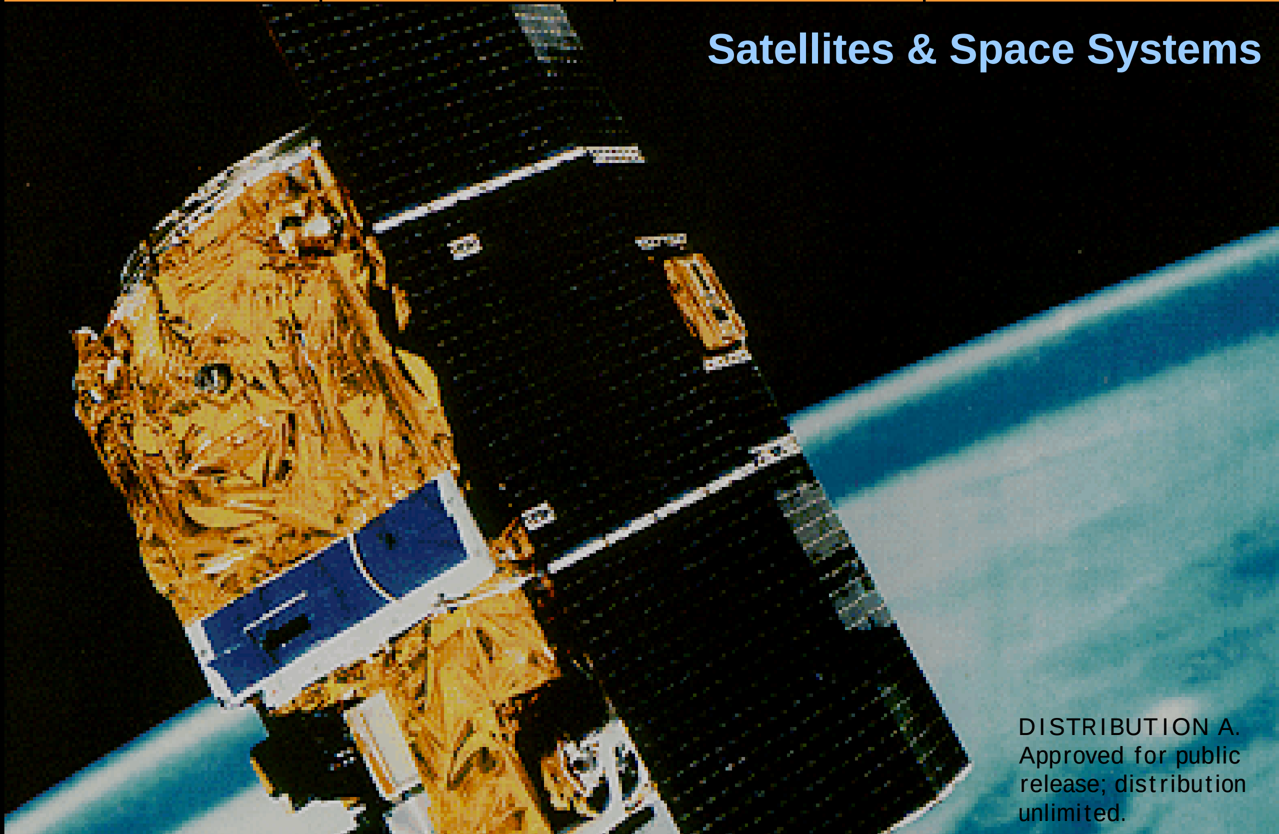
Atomic Oxygen in Lower Earth Orbit

LEO Environment

(Altitudes of 200 to 1500 km)

- Atomic Oxygen (AO): **~10^6 - 10^8 atoms/cm^3**, up to 90 % of the atmosphere at 500km (typical altitude for international space station).
- Typical orbital speed of spacecraft is **7.8 km/sec**
- Actual AO flux on spacecraft **~10^{12} – 10^{14} atoms/cm^2•s**
- **AO Collision energy ~ 5eV** (C-C bond energy ~ 4 eV, C-N ~ 3eV, **Si-O ~ 8.3eV)**
- Low-energy and high energy charged particles.
- Thermal cycling -50 to 150°C
- Solar VUV and UV radiation (~ 100 – 400 nm)
- Bond scission and radical formation can lead to embrittlement.

Bond	Dissociation Energy (eV)	λ (nm)	Material
$-C_6H_4-C(=O)-$	3.9	320	Kapton®
C-N	3.2	390	Kapton®
Si-O	8.3	150	Nanocomposite

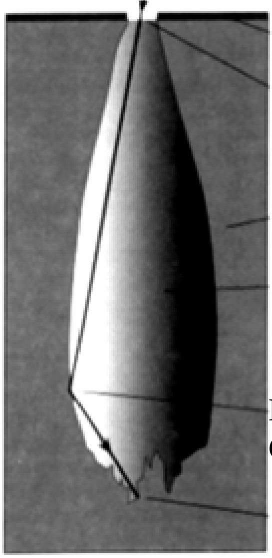


AO Undercutting Study of Aluminized-Kapton MLI
Kim K. de Groh and Bruce A. Banks
Spacecraft and Rockets, Vol. 31, No. 4, (1994)

MLI were flown 5.8 yrs in LEO on the Long Duration Exposure Facility.

Total AO Exposure: 9 x 10^{21} atoms/cm^2
95% of Al-Kapton underwent underpinning.

Oxygen Atom

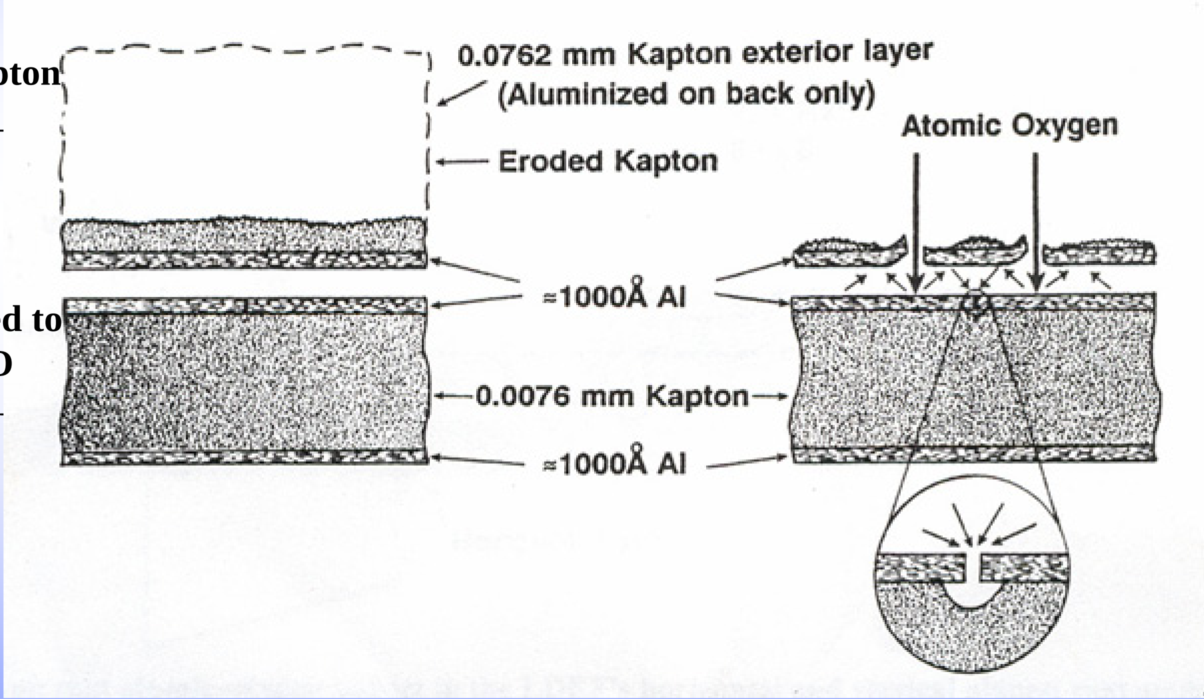


Al Coating
Pinhole Defect
Kapton Substrate
Cavity Eroded By Multiple AO Entries
Nonreactive Collision
Reactive Collision

0.076mm Kapton eroded 2.55x10^{21} O atoms/cm^2

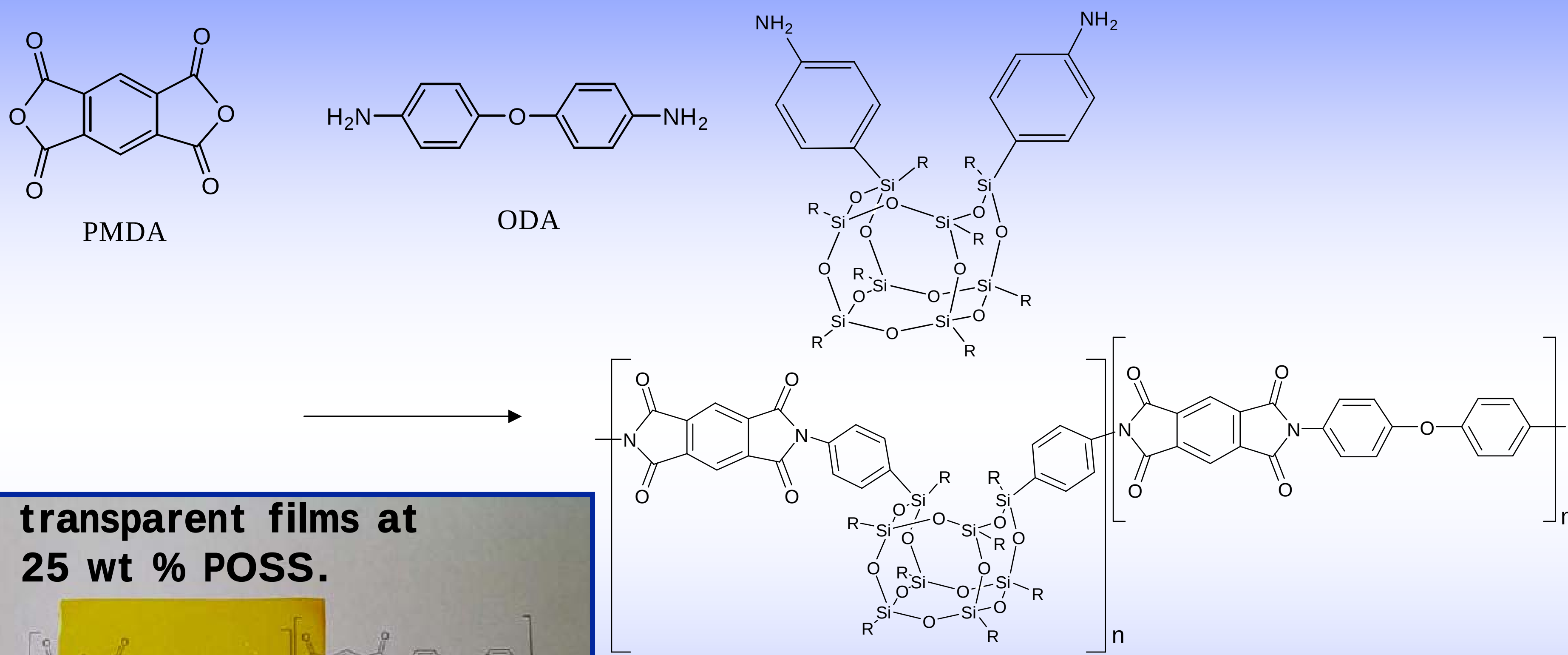
MLI exposed to 6.44 x 10^{21} O atoms/cm^2

Scanning Electron Micrograph Of Kapton MLI Surface.

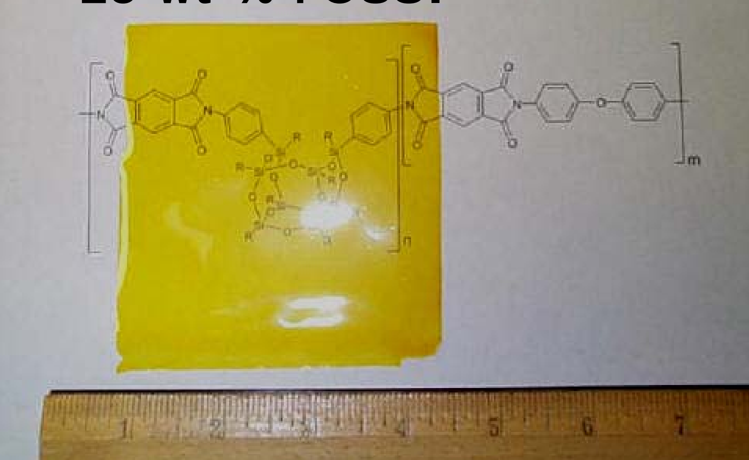


Fluence atoms/cm^3 :
Eroded material divided by
Kapton erosion yield of 3 x 10^{21} cm^3/atom.

POSS-Kapton Polyimides



- **transparent films at 25 wt % POSS.**

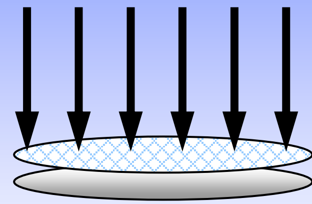


- POSS Polyimides do not lose rigidity above the glass transition temperature .
- Tg of POSS polyimides is 5 – 10 % lower than polyimides (414˚C).
- Room temperature modulus unaffected by POSS.
- High temperature modulus (above 430˚C) is increased with POSS content.

O-Atom Etching Experiment

Total AO fluence of 8.47×10^{20} atoms cm^{-2} (100,000 pulses)

Hyperthermal AO Beam (CO_2 laser, 0 = 4.93-8.42 eV)



Screen

Sample

Kapton H Standard

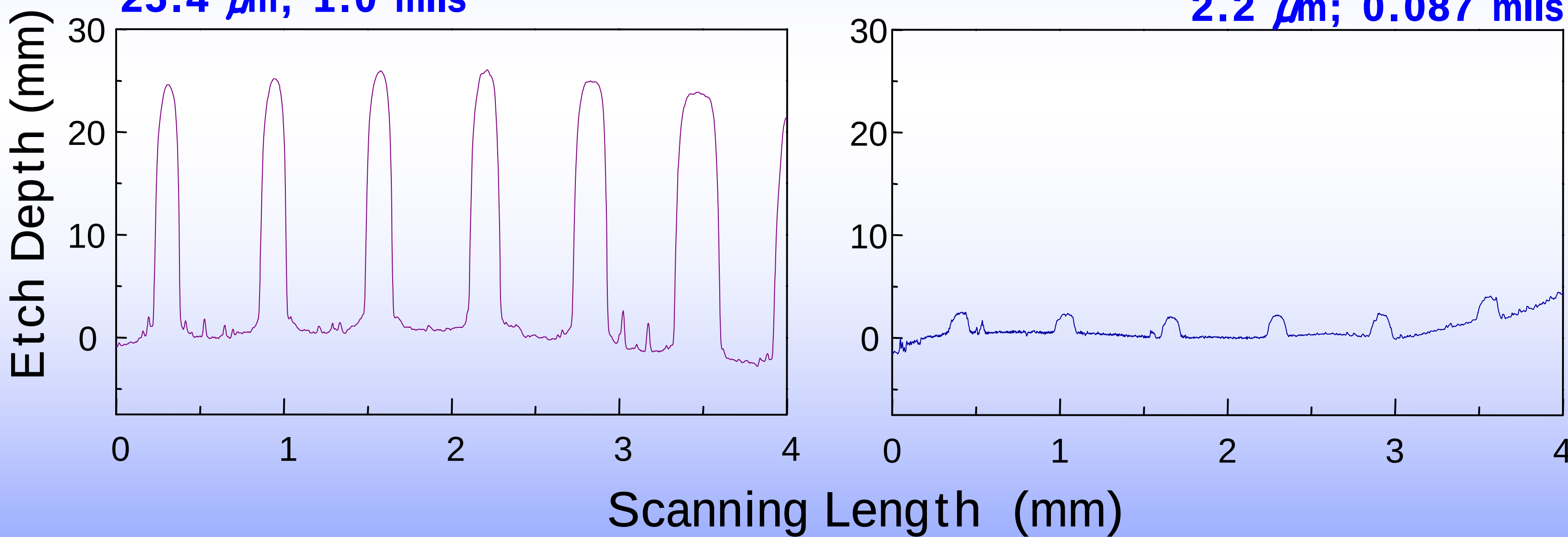
Average etch depth:

25.4 μm; 1.0 mils

Kapton 10 wt% (2 mole %) POSS

Average etch depth:

2.2 μm; 0.087 mils

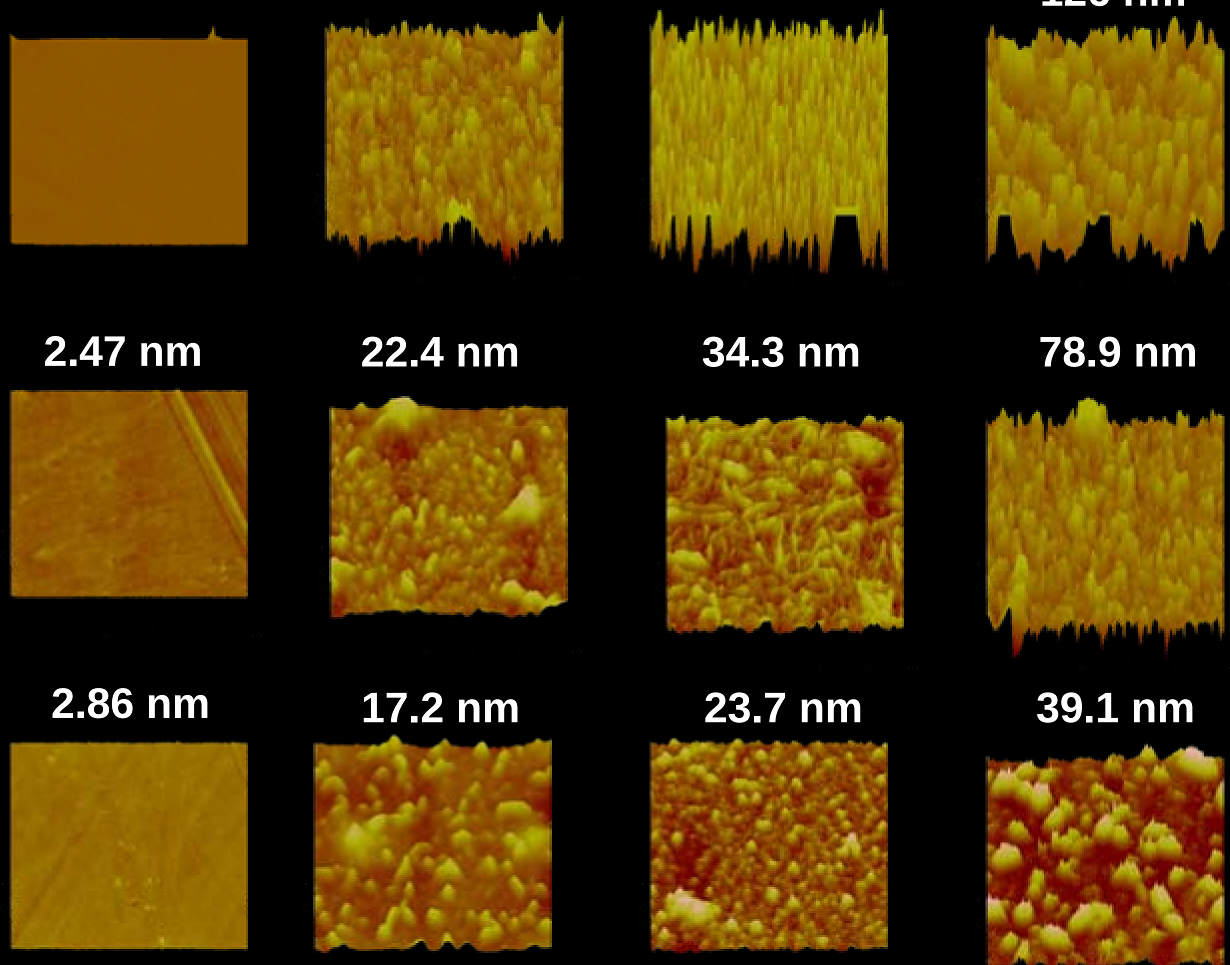


Significantly improved oxidation resistance due to a rapidly formed ceramic-like, passivating silica layer preventing further degradation of underlying virgin polymer.

AFM Images of POSS Polyimides With increasing AO Flux.

(10×10 μm; z scale = 500 nm)

AO fluence (O atoms/cm²) →	0.0 cm^{-2}	3.8×10^{19}	1.6×10^{20}	4.1×10^{20}
0 wt % POSS Polyimide — rms roughness →	2.48 nm	70 nm	120 nm	126 nm
10 wt % POSS Polyimide — rms roughness →	2.47 nm	22.4 nm	34.3 nm	78.9 nm
20 wt % POSS Polyimide — rms roughness →	2.86 nm	17.2 nm	23.7 nm	39.1 nm



Note:

1 x 10^{20} O atoms/cm^2 is roughly equivalent to a spacecraft operating at 500 – 600 km orbit during nominal solar activity conditions for periods of at least 1 year.

Surface Atomic Concentrations (%) determined from XPS (X-ray Photoelectron Spectroscopy) Survey Scans before and after exposure to Atomic Oxygen.

Sample	Exposure (beam pulses)	Kapton-equivalent atomic oxygen fluence (10^{20} O atoms cm^{-2})	C	O	Si	N
0 wt% POSS polyimide	0 6 100 250	0 ~0.1 1.63 4.10	72 69 69 55	19.5 20 24 36	1 2 1 0	7 9 6 9
10 wt% POSS polyimide	0 6 100 250	0 ~0.1 1.63 4.10	77 73 48 **20**	16 18.5 30 **56**	2 5 19 **23.5**	5 3.5 3 0.5
20 wt% POSS polyimide	0 6 100 250	0 ~0.1 1.63 4.10	70 66 20 **12**	20 24 **54** **60**	6 7 **25** **26**	4 3 0 1

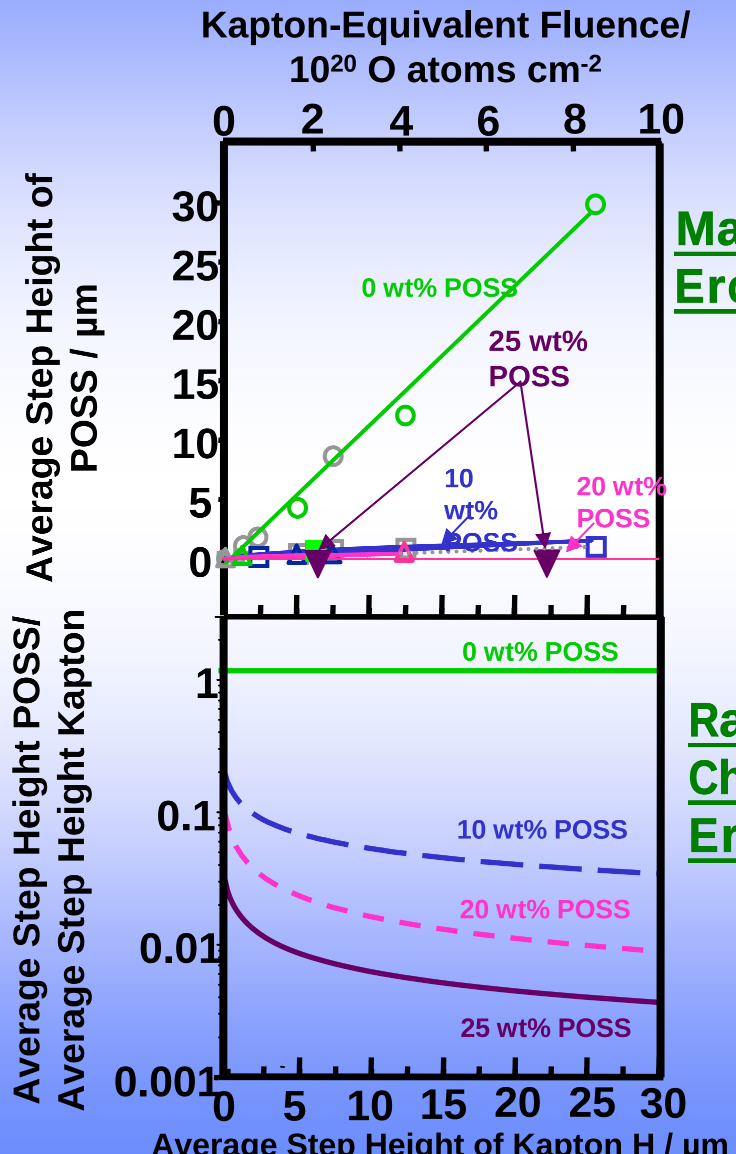
Calculated at%: 0 wt% POSS PI: C = 75.9, O = 17.2, Si = 0, N = 6.9
10 wt% POSS PI: C = 75, O = 17.2, Si = 1, N = 6.4.
20 wt% POSS PI: C = 75, O = 17.2, Si = 1.8, N = 6.0

Erosion of POSS Polyimides by a Beam of Hyperthermal (5eV) O Atoms

1st AO Erosion experiment:
The erosion rate of the 10 and 20 wt % POSS Polyimide samples were 3.7 and 0.98 percent, respectively, of the erosion rate for Kapton H at the highest fluence used in this experiment (8.5×10^{20} atoms cm^{-2}).

2nd AO Erosion Experiment:
The new 25 wt% POSS polyimide samples had an erosion rate that was 0.3 percent of the erosion rate for Kapton H at a fluence of 8.5×10^{20} atoms cm^{-2}.

This erosion rate is one third that of the previously synthesized 20 wt% POSS polyimide.



Material Erosion

Rate of Change in Erosion

Self-passivation of POSS Polyimide Upon Exposure to 2.3×10^{20} O atoms cm^{-2}.

MONTANA STATE UNIVERSITY BOZEMAN

Dr. Timothy Minton
Amy Brunsvold

25 wt % POSS Polyimide

SiO_2 (130 nm) / Kapton (2 mil) / Al (100nm)

Commercial Kapton H

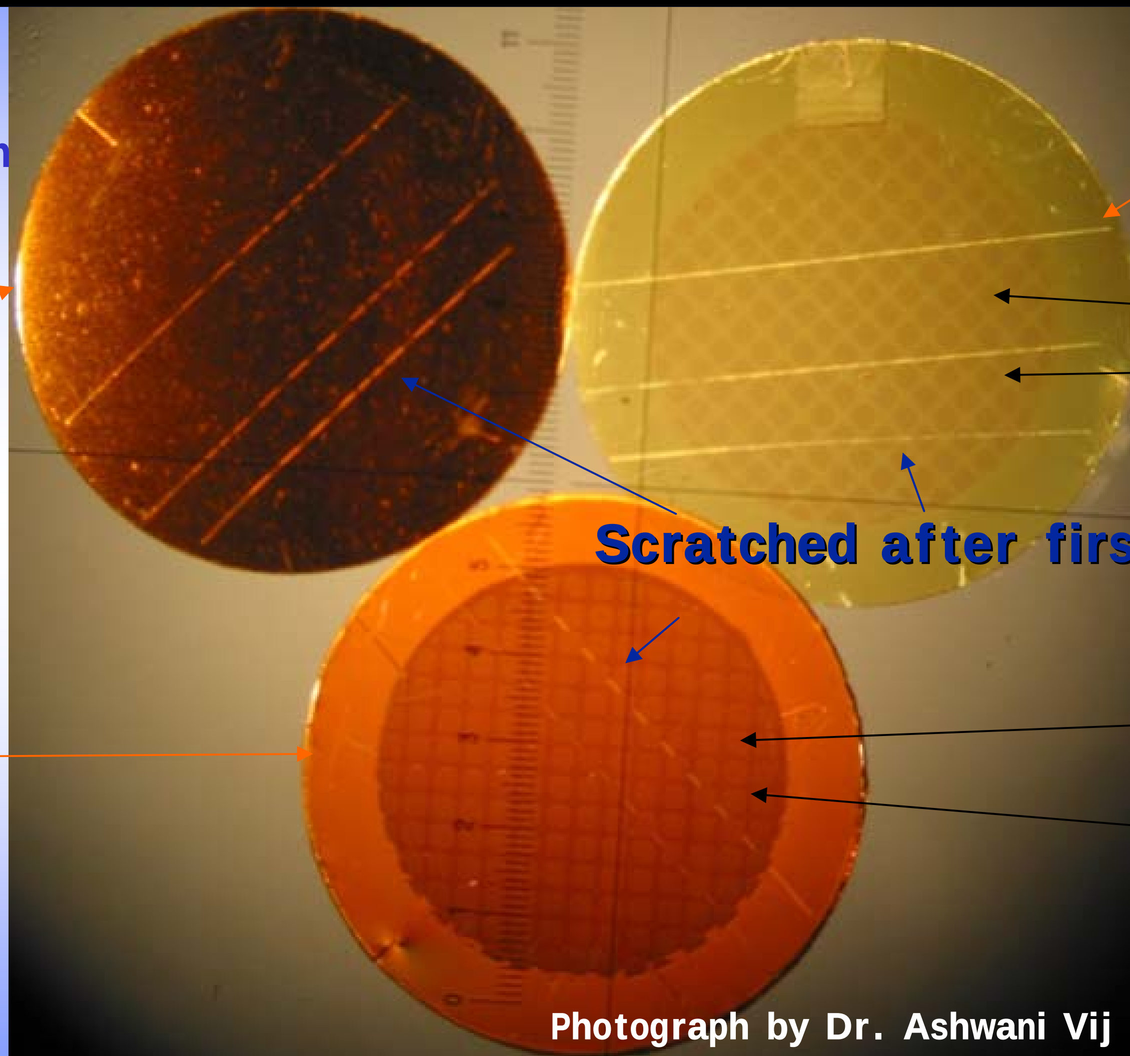
AO Exposed

Protected from AO

Scratched after first AO exposure

AO Exposed

Protected from AO



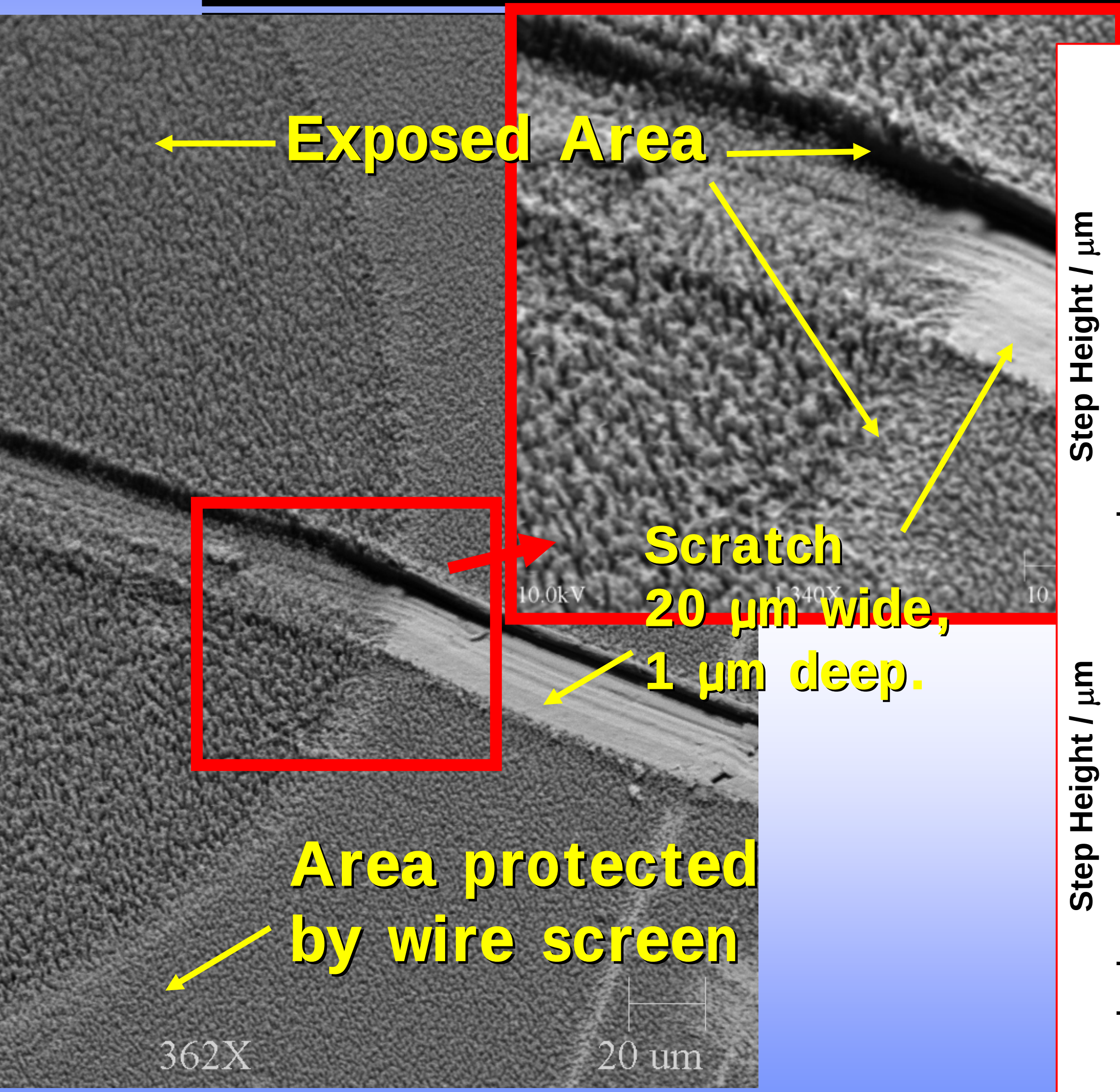
Sample diameter = ½ inch.

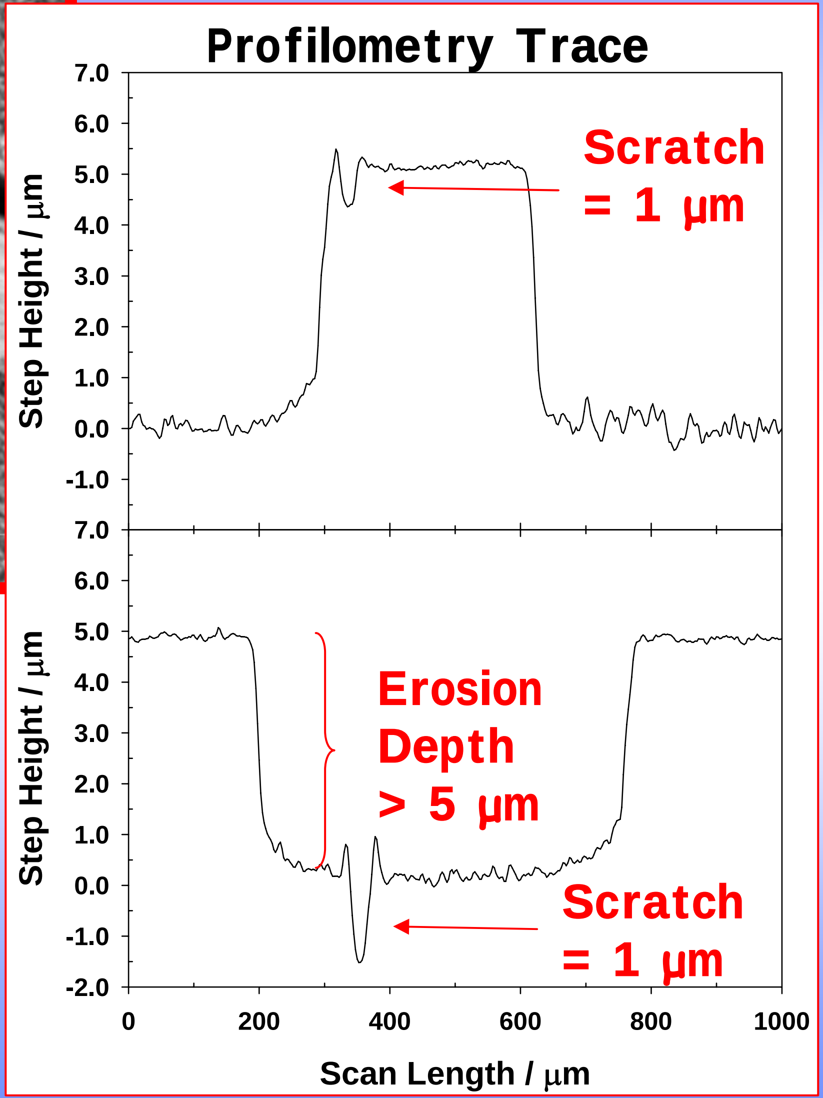
Screen-protected samples were exposed to 2.3×10^{20} O atoms cm^{-2}, unprotected, scratched with a diamond scribe 1 μm deep, screen-protected, and re-exposed to 2.3×10^{20} O atoms cm^{-2}.

Summary of Self-Passivation Study

	SiO_2 coated Kapton HN with Al under-coating	Kapton H	25 wt % POSS polyimide
Erosion depth after 1st exposure to 2.3×10^{20} O atoms cm^{-2}.	~ 0 μm	5 μm	0.200 μm
All samples were scratched 1 μm deep.			
Erosion depth outside of the scratch after 2nd exposure to 2.3×10^{20} O atoms cm^{-2}.	~ 0 μm	5 μm	~ 0 μm
Erosion depth inside of the scratch after 2nd exposure to 2.3×10^{20} O atoms cm^{-2}	7 μm	5 μm	0.200 μm

SEM of Kapton H surface after the Second Atomic Oxygen Exposure

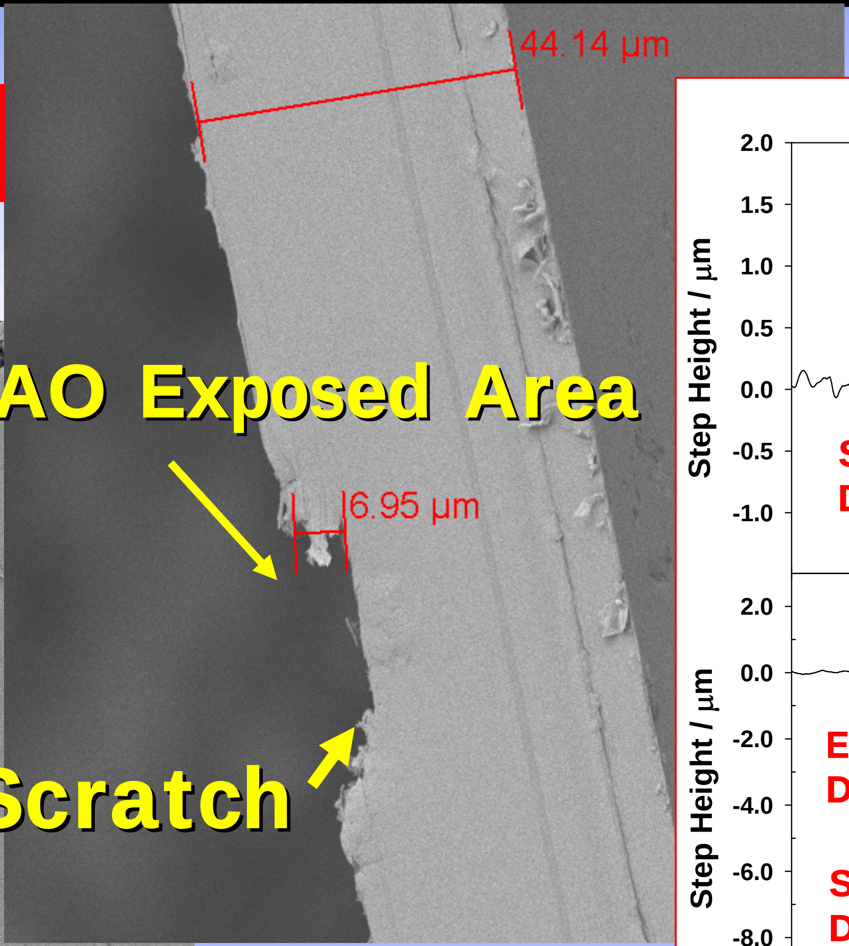


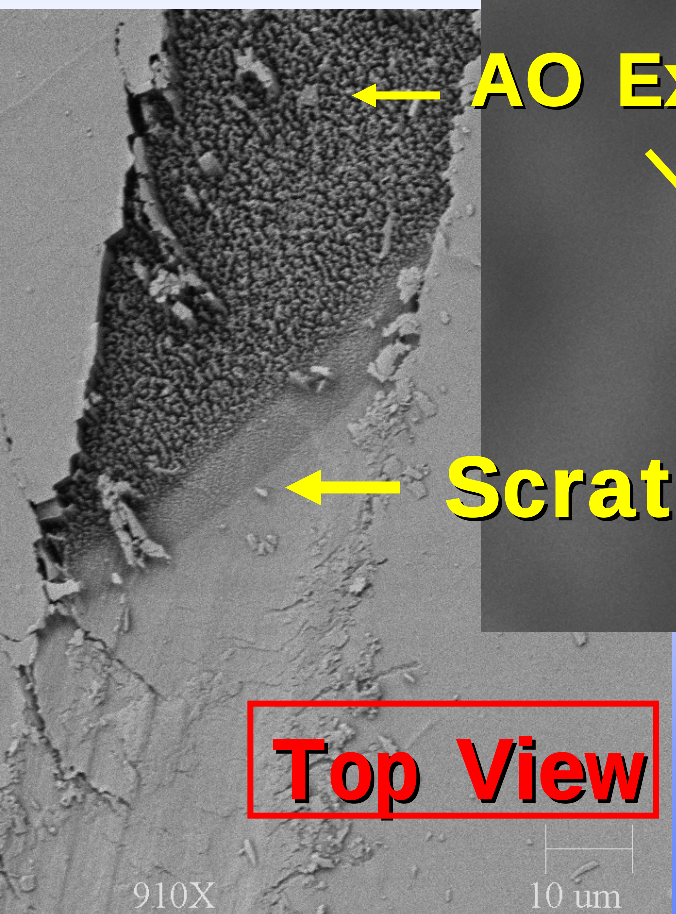


SEM by Marietta Fernandez, Edwards AFRL

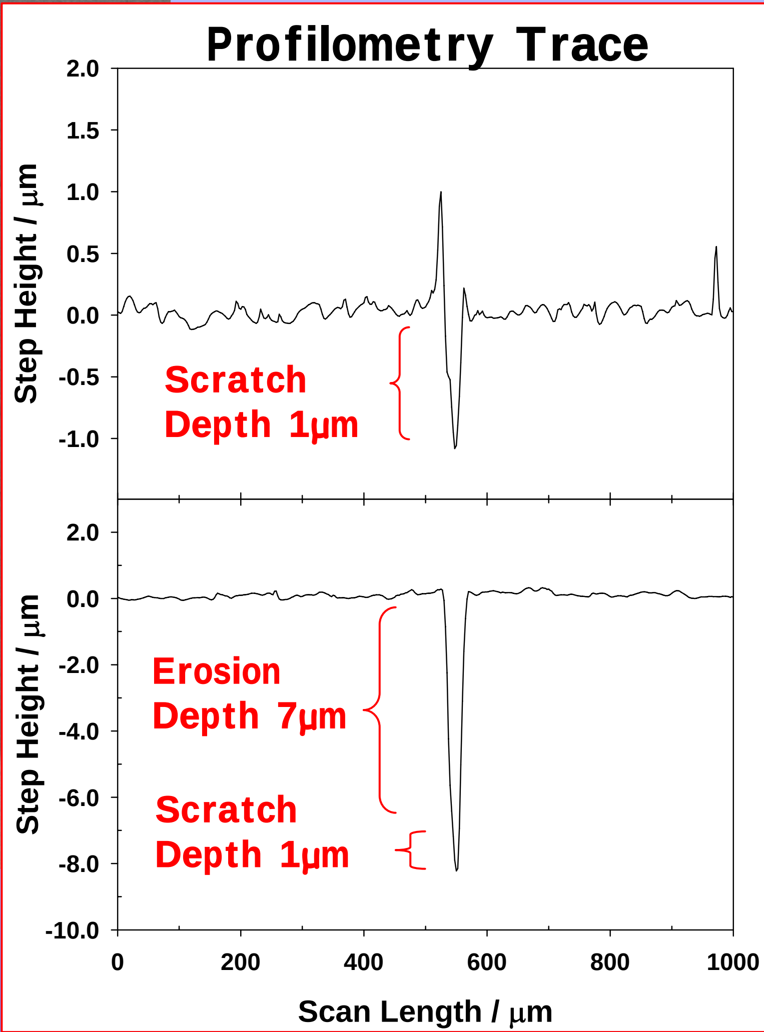
SEM Images of Scratch on SiOx Coated Kapton After Second AO Exposure

Side View

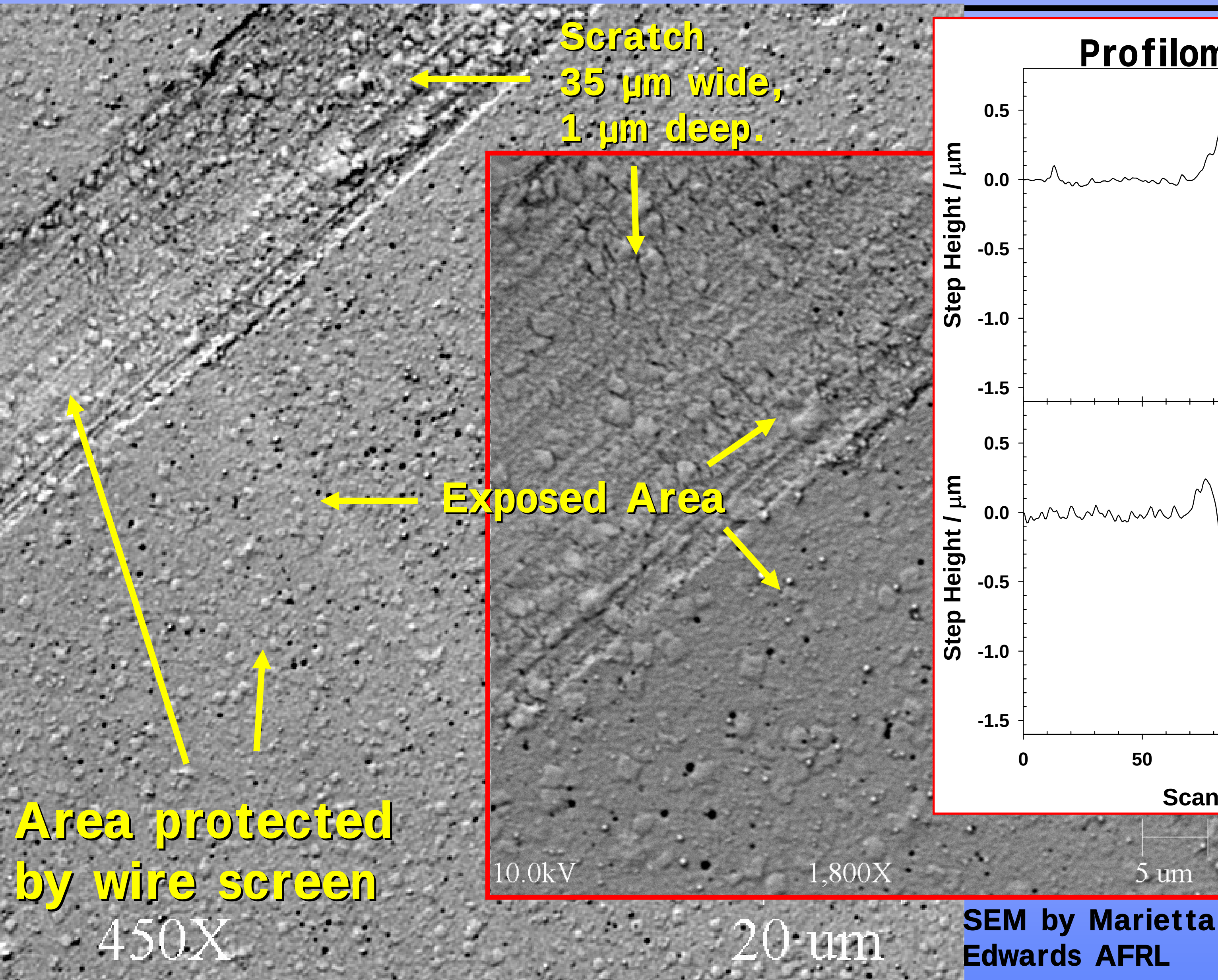


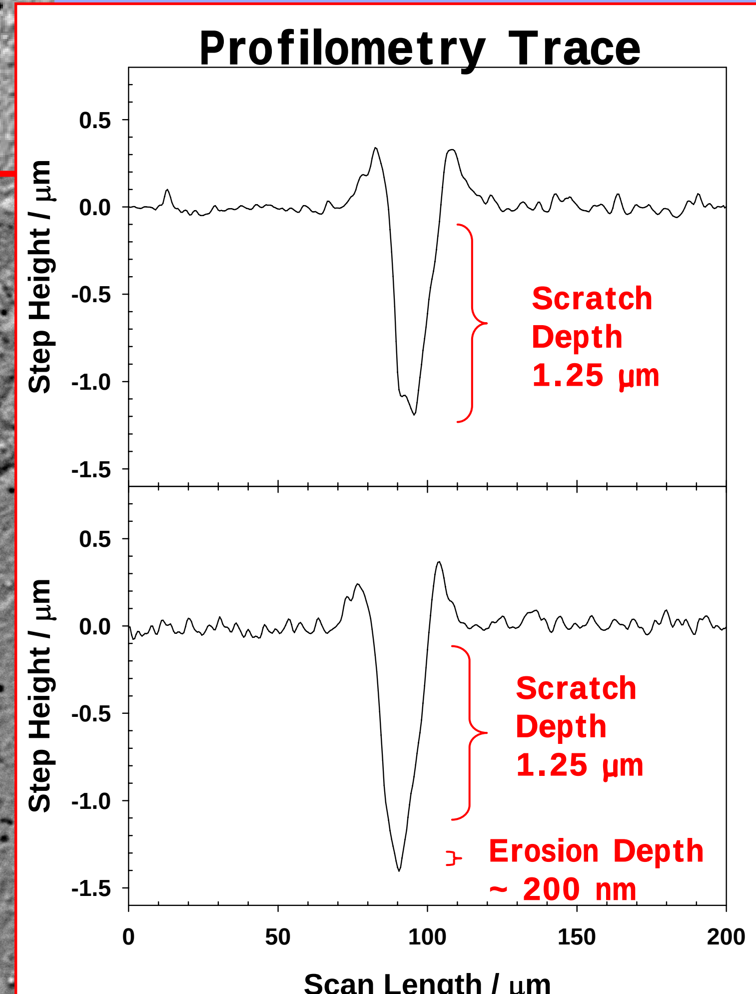


SEM by
Marietta Fernandez
Edwards AFRL



SEM of 25 wt % POSS Polyimide Surface After the Second Atomic Oxygen Exposure





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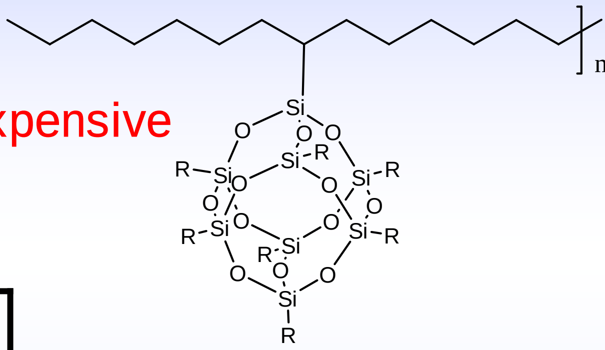
SEM by Marietta Fernandez, Edwards AFRL

New POSS Monomer

NAVAIR

- **China Lake Naval Air Warfare Center, Weapons Division collaborators:**
  Dr. Michael Wright, Dr. Brian Petteys, Dr. Andy Guenthner, Dr. Gregory Yandek.



Recently synthesized:
Side-chain POSS diamine monomer which is relatively inexpensive and of facile synthesis.
Resultant POSS Polyimdes are transparent flexible films.

Sample	Kapton-Equivalent Fluence/10^{20} O atoms cm^{-2}.	Erosion Yield
Kapton H	**4.10**	**12.3**
20 wt % main-chain POSS polyimide	**4.10**	**0.47** **Is 3.8 % of that of Kapton H.**
Kapton H	**3.53**	**10.6**
7 Si8O12 wt % side-chain POSS polyimide.*	**3.53**	**0.35** **Is 3.3 % of that of Kapton H.**

AO resistance is similar between main-chain and side-chain POSS polyimides.

* equivalent SiO content to 20 wt % main-chain POSS PI

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Branch Chief: Dr. Steve Svejda

Collaborators:
Hybrid Plastics: Dr. Joesph Lichtenhan
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